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Low Volatile and Ozone-Depleting Compound Free Solid Film Lubricant

INTERIM REPORT
TFLRF No. 340

by

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Southwest Research Institute
San Antonio, TX

Under Contract to

U.S. Army TARDEC
Petroleum and Water Business Area
Warren, MI 48397-5000

Contract No. DAAK70-92-C-0059

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13. ABSTRACT (Maximum 200 words)

Solid film lubricants are used throughout the Department of Defense (DoD) to meet lubrication requirements in critical weapon system applications. Air cured formulations for Military Specification MIL-L-46147 currently contain solvents such as methyl alcohol, methyl ethyl ketone, and toluene, which are volatile organic compounds (VOC) that are on the Environmental Protection Agency's (EPA) toxic chemical list. The objective of the project was to identify and evaluate replacement lubricants for MIL-L-46147 with reduced overall VOC content. Lubricants that would meet the requirements of MIL-L-46147B, Type II were the focus of this study. Type II lubricants are required to have the low VOC content of 250 g/L. Through industry survey and product testing in the TFLRF (SwRI) laboratory, no products were found to meet all of the specification requirements of MIL-L-46147 Type II. Several lubricants with VOC content below 250 g/L were identified.

14. SUBJECT TERMS

Solid Film Lubricant	Dry Film Lubricant	Lead
Ozone-Depleting Compound	Molybdenum Disulfide	Antimony

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EXECUTIVE SUMMARY

Problems and Objectives: Air-cured formulations of solid film lubricants (SFLs) for Military Specification MIL-L-46147 currently contain solvents such as methyl alcohol, methyl ethyl ketone, and toluene, which are volatile organic compounds (VOC) that are on the Environmental Protection Agency's (EPA) toxic chemical list. These formulations also contain lead and antimony, which are hazardous and potentially carcinogenic. In order to comply with environmental regulations, and to protect the health of personnel, it is necessary to reduce VOC content and eliminate lead and antimony from SFL formulations.

Importance of Project: SFLs are widely used throughout the Department of Defense (DoD) to meet lubrication requirements in critical weapon system applications. This type of lubricant is often used in applications where a liquid lubricant would be difficult to apply, or where contamination from dirt and other particles would be detrimental.

Technical Approach: Candidate formulations were identified and brought to the TARDEC Fuels and Lubricants Research Facility (TARDEC) at Southwest Research Institute (SwRI) in San Antonio, Tx. The main component of this project consisted of laboratory testing and analysis of possible MIL-L-46147B, Type II lubricants. All of the tests required by MIL-L-46147 were performed, with the exception of storage stability. Lead and antimony content was also tested.

Accomplishments: This project resulted in a set of data about commercial products that could be used as MIL-L-46147B, Type II lubricants. While none of the products tested at TFLRF are acceptable for use under MIL-L-46147B, a SFL formulation containing no lead, antimony or VOCs, which meets the military specification, appears feasible. The advancement of pigments and binders is necessary to meet this objective.

Military Impact: The results of this project show the possibility of limiting VOC content and eliminating lead and antimony from SFL formulations.

FOREWORD/ACKNOWLEDGMENTS

This work was performed by the U.S. Army TARDEC Fuels and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, during the period June 1997 through May 1999 under Contract No. DAAK70-92-C-0059. The work was funded and administered by the U.S. Army Tank-Automotive RD&E Center, Petroleum and Water Business Area, Warren, Michigan. Funding was administered by Mr. Tom Landy (AMSTA TR-E/MATR/267), and Mr. Luis Villahermosa (AMSTA TR-D/210) served as the TARDEC contracting officer's representative and project technical monitor.

The authors would like to recognize the technical support provided by J. Dozier and G. Phillips of TFLRF. Special thanks is given to Ms. Wendy Mills of TFLRF for her help in the preparation and editing of this report.

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SYMBOLS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
CRDA	Cooperative Research and Development Agreement
DoD	Department of Defense
EPA	Environmental Protection Agency
MSDS	Material Safety Data Sheet
MoS_2	Molybdenum Disulfide
OSHA	Occupational Safety and Health Administration
SFL	Solid Film Lubricant
SwRI	Southwest Research Institute
TFLRF	U.S. Army TARDEC Fuels and Lubricants Research Facility
VOC	Volatile Organic Compound

1.0 BACKGROUND AND INTRODUCTION

Solid film lubricants (SFLs) are widely used throughout the Department of Defense (DoD) to meet lubrication requirements in critical weapon-system applications. This type of lubricant is often used in applications where a liquid lubricant would be difficult to apply, or where contamination from dirt and other particles would be detrimental. SFLs can be used as a sacrificial start-up lubricant, or as a long-term lubricant over the life of the part. Many solid film lubricants also offer long-term protection against corrosion.

Air-cured formulations of SFLs for Military Specification MIL-L-46147 currently contain solvents such as methyl alcohol, methyl ethyl ketone, and toluene, which are volatile organic compounds (VOC) that are on the Environmental Protection Agency's (EPA) toxic chemical list. A VOC is defined to be any compound of carbon, (excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and ammonium carbonate) which participates in atmospheric photochemical reactions. (1)*

In order to comply with environmental regulations, it is necessary to reduce VOC content. MIL-L-46147B contains a specification for a Type II lubricant that has a VOC content of 250 g/L or less. In MIL-L-46147B, the Type II lubricants are allotted a longer drying time and have a slightly shorter endurance life requirement than the Type I lubricants with high VOC content.

In addition to the VOC content, current SFLs contain lead and antimony. The EPA classifies lead and lead compounds as an Extremely Hazardous Substance, and the Occupational Safety and Health Administration (OSHA) has identified antimony compounds as potential carcinogens. These chemicals make the application and disposal of SFLs hazardous.

The objective of this project was to identify and evaluate replacement lubricants for MIL-L-46147B Type II. The test procedures and requirements contained in the military specification were used as the guideline for testing.

*Numbers in parentheses indicate references at the end of the document.

2.0 APPROACH

The first step for this project was to identify potential products. SFL manufacturers with whom Cooperative Research and Development Agreements (CRDAs) had formerly been established were contacted first and asked to provide samples of any products suitable for MIL-L-46147 Type II. Also, the Thomas Register was used to identify other manufacturers of SFLs. This search resulted in a large number of contacts, and those manufacturers were contacted with a form letter requesting information on any suitable products. These efforts yielded several products that were predicted to meet the VOC limitation as well as the other specifications.

Eight lubricant samples were received at the TARDEC Fuels and Lubricants Research Facility (TFLRF) at Southwest Research Institute (SwRI) in San Antonio, TX for testing (Table 1). All of the tests required by MIL-L-46147B were performed, with the exception of storage stability. Test procedures are described in Section 3. Results are presented in Section 4, and Section 5 contains discussion and conclusions. Table 1 lists the eight samples tested. Note that samples SFL-7 and SFL-8 are identical compounds except for the different cure procedures. This is also the case with samples SFL-9 and SFL-10.

Table 1. Products Tested

Name	AL Code	Manufacturer	Product
SFL-1	AL-25354	McGee (Air Cured)	MoS2-900
SFL-2	AL-25355	McGee (Air Cured)	MoS2-108L
SFL-3	AL-25356	McGee (Air Cured)	MAC-544
SFL-4	AL-25413	Sandstrom (Air Cured)	MoS2
SFL-5	AL-25420	Tiodize (Air Cured)	Tiolube 75/75
SFL-6	AL-25435	E/M (Air Cured)	Lubribond K
SFL-7	AL-25471	CFI (Air Cured)	Alseal 380
SFL-8	AL-25471	CFI (Heat Cured)	Alseal 380
SFL-9	AL-25472	CFI (Air Cured)	Urethabond W119U
SFL-10	AL-25472	CFI (Heat Cured)	Urethabond W119U

3.0 TEST PROCEDURES

MIL-L-46147B requires that SFLs pass numerous tests related to the following film properties: lubricant content; endurance life and load carrying capacity; and resistance to fluids, thermal shock, and corrosion. The following subsections describe those tests, pass/fail criteria, and any modifications or deviations to the procedure that were made. Table 2 contains a brief summary of the test procedures.

3.1 Film Appearance

According to MIL-L-46147, the bonded SFL must be subjected to a visual inspection and found to be uniform in color and smooth in appearance. The film also must be free from cracks, sags, foreign matter, grit, rough particles, or separation of ingredients.

These visual inspections of film appearance were performed on each sample by two individuals.

Table 2. Test Procedure Summary

Test	Method	Pass Criteria
Film Appearance	Visual Inspection	No cracks, sags, foreign matter, grit, rough particles, or ingredient separation
Film Thickness (aluminum)	ASTM D 1400 or B 244	0.008 to 0.013 mm, with no measurement less than 0.005 or greater than 0.018
Film Thickness (steel)	ASTM D 1186 or B 499	0.008 to 0.013 mm, with no measurement less than 0.005 or greater than 0.018
Film Adhesion	ASTM D 2510 A	Lubricant does not lift to expose bare surface
Fluid Resistance	ASTM 2510 C	Lubricant does not lift to expose bare surface
Endurance Life	ASTM D 2625 A	Average of 90 min.; no test less than 75 min
Load Capacity	ASTM D 2625 C	Average of 2500 lbf; no test less than 2000 lbf
Corrosion Protection	ASTM B 117	No more than 3 rust spots; none bigger than 1 mm diameter
Thermal Shock Sensitivity	ASTM D 2511	Lubricant does not crack, flake, soften, or fail film adhesion test
Solids Content	MIL-L-46147, 4.6.3.1	Greater than 24%
Volatile Organic Compound Content	ASTM D 3960	Less than 250 g/L
Storage Stability	MIL-L-46147 , 3.13	After one year, pass all tests except solids content

3.2 Film Thickness

The thickness of the dry lubricant must be between 0.008 and 0.013 mm, with no reading less than 0.005 mm or greater than 0.018 mm. Measurements taken on steel panels were performed according to ASTM D 1186. Measurements taken on aluminum panels were performed according to ASTM D 1400.

ASTM D 1186 was used to measure the lubricant thickness on the steel panels. A permanent magnet was used for this test method. The device was calibrated using shims of known thickness, then used to measure lubricant film thickness. There was one deviation from this procedure. ASTM D 1186 requires that the thickness of the shims be known to $\pm 0.5 \times 10^{-7}$ m. However, the thickness of the shims that were used was known only to $\pm 1.0 \times 10^{-7}$ m. The reported thickness for a test specimen was the average of ten measurements for that specimen.

ASTM D 1400 was used to measure lubricant thickness on the aluminum panels. This test method requires an eddy current thickness gage. The gage would be calibrated with shims of known thickness, then used to measure the lubricant film thickness. However, (as described in the previous paragraph) the thickness of the shims was known only to $\pm 1.0 \times 10^{-7}$ m, instead of $\pm 0.5 \times 10^{-7}$ m as required. Also, Section 5.1 of ASTM D 1400 states that the test apparatus would be "Eddy Current Thickness Gages suitable to measure coating thickness accurately." The standard test apparatus used was not commercially available. The apparatus used was a combination of three instruments purchased separately. The apparatus was proven to accurately measure coating thickness within ASTM specifications. After calibration, the repeatability of the apparatus was tested and met the requirements described in the ASTM test method. The reported film thickness for each test specimen was the average of five measurements taken from various locations on that specimen.

3.3 Film Adhesion

The adhesive properties of the lubricant must be tested under ASTM D 2510 Procedure A. This test requires that an aluminum panel coated with the lubricant be immersed in water for 24 hours and wiped dry. A piece of masking tape is then pressed onto the surface of the lubricant and abruptly pulled off. If

the lubricant does not lift to expose any bare surface, the test is considered successful. There were no modifications to this procedure.

3.4 Fluid Resistance

The lubricant's resistance to different fluids must be tested under ASTM D 2510 Procedure B. This test requires that an aluminum panel coated with the lubricant be immersed for 24 hours in each of the 11 fluids listed in MIL-L-46147. The panel is then rinsed and air dried, and a piece of masking tape is pressed onto the surface. If no lubricant is lifted to expose any bare surface when the tape is pulled off, the test is considered successful. This procedure was followed with no modifications.

3.5 Endurance Life

The endurance life of the lubricant, when tested according to ASTM D 2625 with a gauge load of 1000 lbf, must average at least 90 minutes, with no single test less than 75 minutes. This test procedure requires applying the lubricant to four sets of Falex pins and vee-blocks. A Falex pin and vee-block test machine is then used to rotate the pin within the vee's of two of the vee-blocks. Endurance life is the elapsed time from the start of the test until the torque between the pin and the vee-blocks increases by 10 in.lbf.

This test procedure was followed with one modification. It is stated in MIL-L-46147B Section 3.5 that this test must be performed on phosphated steel specimens with lubricant applied to a cured film thickness of 0.008 to 0.013 mm. However, due to the unusual geometry of the vee-blocks and pins, thickness measurements would have been difficult to obtain. Considerable time and expense would have been required to modify the permanent magnet device to make it suitable for those measurements. Therefore, it was assumed that the coating thickness on the pins and vee-blocks was similar to the coating thickness on flat phosphated steel panels that were (1) coated at the same time as the pins and vee-blocks, (2) coated with the same lubricant, and (3) coated by the same operator.

3.6 Load Carrying Capacity

The load carrying capacity of the lubricant, when tested according to ASTM D 2625, must average at least 2500 lbf with no single test less than 2000 lbf. This procedure also uses the Falex Pin and Vee-Block Test Machine. The maximum load capacity of the lubricant is the greatest load that the lubricant can sustain for at least one minute without a sudden increase in torque between the pins and vee-blocks.

This test procedure was followed with two modifications: coating thickness was not measured before the test was performed (described in Section 3.4 Endurance Life); and test repetitions were reduced from four (as required by the ASTM test method) to two. MIL-L-46010, "Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting," contains an almost identical set of test procedures to MIL-L-46147. It states in MIL-L-46010 that load carrying capacity must be tested in at least two trials.

3.7 Corrosion Protection (Salt Spray)

The corrosion resistance of the lubricants was tested according to ASTM B 117. This test requires that a phosphated steel panel be placed in a humidity cabinet and exposed to a 5% salt spray for 100 hours. Passage of the test requires that no more than three rust spots per panel be found, and that no rust spot be greater than 1 mm in diameter. This test procedure was followed with no modifications.

3.8 Thermal Shock Sensitivity

The lubricant's sensitivity to thermal shock was tested according to ASTM D 2511. This test requires placing an aluminum panel coated with SFL in an oven at 191°C for three hours, then immediately transferring it to a subzero cabinet at -54°C for three hours. The panel is then examined. The lubricant would fail the test if it flaked, cracked, softened, or did not pass a film adhesion test.

This test was performed with one modification. A subzero cabinet at -54°C was not available; a subzero cabinet at -50°C was substituted. It is believed that this modification did not impact the results.

3.9 Solids Content

The total solids content of each lubricant was tested according to the procedure in Section 4.6.3.1 of MIL-L-46147. Under this procedure, the weight of a sample of bulk lubricant was measured before and after being kept for 18 hours in an oven at 49°C. Based on the change in weight, the percent of solids in the lubricant formulation was calculated. Lubricants must contain at least 24% solids to meet the specification.

3.10 Volatile Organic Compound Content

MIL-L-46147 states that the VOC content of Type II lubricants must be less than 250 g/L. An ASTM test for determining VOC content is given in the specification. However, since the information can also be obtained from the Material Safety Data Sheet (MSDS) of the lubricant, testing was not performed.

3.11 Lubricant Application

Many of the tests described in Sections 3.1 through 3.10 require the use of metal specimens coated with the SFL being tested. It has been found that the quality of the pretreatment of the specimens and the application of the lubricant can be pivotal factors in the lubricant's performance. (2,3)

Pretreatment of the specimens varied depending on the type of metal being used. Aluminum panels conforming to QQ-A-250/5 were anodized and sealed in accordance with MIL-A-8625 Type II, sulfuric acid anodize. Steel panels conforming to ASTM A 108 were sandblasted with aluminum oxide and then phosphated in accordance with DOD-P-16232, zinc phosphate. This pretreatment was applied to the steel pins and vee-blocks as well as the panels.

Prior to applying any lubricant, the panels were cleaned with Aliphatic Naptha conforming to TT-N-95. After being cleaned, each specimen was tested according to ASTM F 22 to ensure a clean surface. If the specimen did not pass ASTM F 22, it was recleaned and retested.

The specimens were air dried, then coated with lubricant using a Devilbiss EGA spray gun with the smallest nozzle available. All aluminum and steel specimens were coated in a single batch. The specimens were then allowed to dry for a minimum of 24 hours before any testing or measurements were performed on them.

4.0 RESULTS

Four samples of low VOC content SFLs for potential use under MIL-L-46147B are listed in Table 3. These samples met the specification requiring low VOCs and are tested with the other SFL samples. These lubricants differ from the other samples in that they are either water based or have a very low volatile component.

Table 3. MIL-L-Low VOC Content Lubricants

Lubricant	AL Code	Manufacturer	Product	VOC Content (g/L)
SFL-1	AL-25354	McGee	MoS2-900	0
SFL-4	AL-25413	Sandstrom	MoS2	66
SFL-7	AL-25471	CFI	Alseal 380	0
SFL-9	AL-25472	CFI	Urethabond W119U	69

The lubricant pigment of each lubricant tested was molybdenum disulfide (MoS_2). SFL-1 is a water-based lubricant from McGee Industries with one hazardous ingredient: silicic acid. It contains no VOCs. SFL-4 is a water-based lubricant from the Sandstrom Products Company that includes the hazardous ingredient dimethylethanolamine. It has a very low VOC content. SFL-7 and SFL-9 are both from Coatings for Industry, Inc (CFI). Both CFI lubricants meet the VOC limit. SFL-7 contains a small amount of N-methyl-pyrrolidone, which is volatile; SFL-9 contains alkali metal silicate, which is hazardous. (4) SFL-9 is an experimental lubricant that has recently been developed by CFI with a very strong binder.

The tests described in Section 3 were performed on each lubricant in Table 4, which contains a summary of the test results detailed in the following subsections. Note that results are not available for all of the SFL-5 tests. The manufacturer of this lubricant did not supply TFLRF with a sample of the lubricant. Instead, uncoated specimens were sent from TFLRF to the manufacturer to be coated with lubricant and then returned. All other SFLs were coated at TFLRF.

Table 4. Test Results					
Lubricant	**Film Appearance**	**Film Thickness**	**Solids Content**	**Film Adhesion**	**Fluid Resistance**
SFL-1	Pass	0.00689	10.32	Pass	Fail
SFL-2	Pass	0.00634	9.30	Pass	Pass
SFL-3	Pass	0.00929	4.55	Pass	Fail
SFL-4	Pass	0.02591	45	Pass	Pass
SFL-5	Pass	0.00701	N/A*	Pass	Pass
SFL-6	Pass	0.00957	11	Pass	Pass
SFL-7	Pass	0.02363	51.77	Pass	Pass
SFL-9	Pass	0.02282	32.77	Pass	Pass
Lubricant	**Thermal Shock Sensitivity**	**Endurance Life**	**Load Capacity**	**Corrosion Protection**	
SFL-1	Pass	12:14	1875	Fail	
SFL-2	Pass	7:23	1125	Fail	
SFL-3	Pass	**	**	Fail	
SFL-4	Pass	2:31	1000	Fail	
SFL-5	Pass	3:41	825	Fail	
SFL-6	Pass	**	**	Fail	
SFL-7	Pass	21:10	1470	Fail	
SFL-9	Pass	5:44	1320		

*Unable to perform specified test because the manufacturer did not supply the lubricant in bulk.
** These tests were not conducted because the samples had failed several other tests and were not considered acceptable.

4.1 Film Appearance

The film of each candidate lubricant was found to be free from cracks, sags, foreign matter, grit, and rough particles, and each was uniform in color as determined by visual inspection. Therefore, the films were acceptable based on the requirements listed in Section 3.1.

4.2 Film Thickness

Three aluminum panels and two steel panels were chosen from the set of coated panels. The steel panels were tested under ASTM D 1186 for film thickness with a permanent magnet, and the aluminum panels were tested under ASTM D 1400. Five areas on each aluminum panel were measured for thickness, and

ten spots were measured on each steel panel, totaling 35 measurements per lubricant. The reported thickness is the average of the 35 measurements. One coat was applied for all products except SFL-3, which required three coats. SFL-4, SFL-7, and SFL-9, could not be thinned sufficiently to meet the thickness specification. These products could only be considered for use in applications where film thickness is not critical. Results are presented in Table 4 and Appendix B.

4.3 Solids Content

The solids content of each product was tested with the method described in Section 4.6.3.1 of MIL-L-46147. The requirement of 24% residue after heating for 18 hours was met only by SFL-4, SFL-7, and SFL-9. The rest of the samples were considered a fail based on the results presented in Table 4.

4.4 Film Adhesion

The film adhesion of each product was determined by the method described in Section 3.3. All of the products passed, i.e., after 24 hours of immersion in water, none of the film was lifted with the application of masking tape.

4.5 Fluid Resistance

The fluid resistance of each product was determined by the method described in Section 3.4. SFL-1 and SFL-3 failed with all 11 fluids. The remaining products passed the tests with all 11 fluids. The 11 fluids consisted of water, solvents, lubricants and fuels and represent typical fluids the lubricant would be exposed to during service.

4.6 Thermal Shock Sensitivity

The thermal shock sensitivity of each product was determined by the method described in Section 3.8. None of the films flaked, cracked or softened, and all passed the film adhesion test after removal from the subzero cabinet. Therefore, all products passed this test.

4.7 Endurance Life

The endurance life of each product was determined by the Falex pin and vee-block method described in Section 3.5. The data are presented in Table 4 and Appendix C. The results listed in Table 4 show that the longest life observed during the pin and vee-block testing was 21.10 minutes, far less than the required minimum of 75 minutes.

4.8 Load Capacity

The load carrying capacity of each product was determined by the method described in Section 3.6. No lubricants met the 2500 lbf minimum. Data presented in Table 4 and Appendix C show that the greatest load capacity was 1,875 lbf for SFL-1; the remaining were considerably less.

4.9 Corrosion Protection

The corrosion protection of each product was determined by the salt spray method described in Section 3.7. At the end of the test, all of the panels were more than 10% covered with rust . Passing this test requires no more than three rust spots with none larger than 1 mm. Therefore, all of the products failed this test.

5.0 CONCLUSIONS

Molybdenum disulfide (MoS_2) dry film lubricants are not acceptable replacements for the current lead and antimony solid film lubricants (SFLs). MoS_2 products do not provide adequate endurance life or load carrying capacity. Lead and antimony combine synergistically, producing a more effective lubricant than a MoS_2 coating. Furthermore, molybdenum disulfide films do not successfully inhibit corrosion. Dibasic lead phosphate provides corrosion protection by absorbing acids before they come in contact with lubricated parts.

Overall, the absence of lead and antimony results in decreased lubricant effectiveness and durability. While none of the lubricants tested at the TARDEC Fuels and Lubricants Research Facility (TFLRF) are acceptable for use under MIL-L-46147B, a SFL formulation containing no lead, antimony or vola-

tile organic compounds (VOCs), which meets the military specification, appears feasible. The advancement of pigments and binders is necessary to meet this objective.

Products using solid silicon compound binders were superior to other products tested for endurance life and load capacity. Other binders exist that may provide more effective wear resistance. More research with binding materials (such as these silicon compounds) must be conducted before concluding that these compounds increase endurance life or load carrying capacity.

SFL-7 is 8% alkali metal silicates, which is most likely a combination of potassium silicate and sodium silicate. It provides the most effective lubrication of the products tested, with an endurance life of over 22 minutes. Potassium silicate is a soluble potash glass with the following chemical formulas: K_2SiO_3, $K_2Si_2O_5$, and K_4SiO_4. This chemical is typically found in the form of slightly soluble glass-like pieces. It is used as a binder in the manufacturing of glass and protective coatings such as SFLs. Sodium silicate is a water glass with the following formulas: Na_2SiO_3, $Na_6Si_2O_7$, and $Na_2Si_3O_7$. Sodium silicate is also a slightly soluble crystal found in pieces or lumps. It is used to line Bessemer converters.

SFL-1 contains 1 to 4% silicic acid, H_2SiO_3. Silicic acid is a colorless-to-gray powder or solid from precipitated silica (found in nature as opal). This powder is soluble in hot alkaline hydroxide solutions.

The silicic acid and alkali metal silicate's main purpose is to bond the lubricant to the coated surface. The alkali metal silicates are also thought to enhance corrosion protection by increasing the hydroxide ion concentration, thus preventing acid from reaching the coated surface.

MoS_2 is the pigment used by all of the products tested at TFLRF. Product literature indicates that tungsten disulfide (WS_2) is a more effective lubricant. Tungsten is harder than molybdenum, indicating that its endurance life is greater. Tungsten also resists attack by a great number of compounds found in nature. The main drawback of tungsten disulfide is its cost, which is nearly four times greater than molybdenum disulfide's published price.

The VOC content of a SFL can easily be decreased or eliminated by using water as a solvent, as in SFL-1, SFL-4, SFL-7 and SFL-9. However, some binders are insoluble in water, therefore an alternative solvent that could be used as a VOC spraying medium is carbon dioxide (CO_2). CO_2 is currently being used to eliminate VOCs in the spray application of paint. CO_2 would need to be pressurized to at least five atmospheres in order for it to become liquid. This pressurization would allow the lubricant to be dispersed in the same medium in which it is contained, with no other pressurization required. See Appendix E for more information on liquid carbon dioxide.

6.0 REFERENCES

1. Federal Register, Volume 62 No. 164, Monday, August 25, 1997, Pages 44900-44903.

2. TARDEC Letter Report 97-3, "Elimination of Lead, Antimony, and CFC Compounds from Military Specification Low VOC Solid Film Lubricants," April 1997.

3. E/M Corporation Report 3000-A, "Recommended Application Procedure for Solid Film Lubricants and Coatings," September 1990.

4. United States Environmental Protection Agency Report 745B95002, "Emergency Planning and Community Right to Know Section 303: Toxic Chemicals List," May 1995.

APPENDIX A
COMPOSITION

Table A-1. Product Composition						
Identification	**Component**	**Percent**	**EPA List**	**ODC**	**VOC**	**CAS#**
SFL-1	Silicic acid, sodium salt	1-4	No	No	No	1344-09-8
	Molybdenum Disulfide	10-15	No	No	No	1317-33-5
	Water	80-85	No	No	No	7732-18-5
SFL-2	Heptane	40-45	No	No	Yes	142-82-5
	Ethyl Alcohol	15-20	No	No	Yes	64-17-5
	Butyl Acetate	15-20	No	No	Yes	123-86-4
	Molybdenum Disulfide	15-20	No	No	No	1317-33-5
SFL-3	Molybdenum Disulfide	3-7	No	No	No	1317-33-5
	Isoparaffinic Hydrocarbon	40-50	Possible	No	Yes	64742-48-9
	2-Propoxyethanol	40-50	No	No	Yes	2807-30-9
SFL-4	Molybdenum Disulfide	N/S	No	No	No	1317-33-5
	Demethylethanolamine	N/S	Yes	No	Yes	108-01-0
	Water	N/S	No	No	No	7732-18-5
SFL-5	Molybdenum Disulfide	N/S	No	No	No	1317-33-5
	Resin	N/S	Possible	No	Yes	Mixture
	Sb203 (Antimony)	N/S	Yes	Yes	No	1309-64-4
SFL-6	Molybdenum Disulfide	N/S	No	No	No	1317-33-5
	Methyl Ethyl Ketone	75-85	Yes	No	Yes	78-93-3
	Xylene	<10	Yes	No	Yes	1330-20-7
SFL-7	Alkali Metal Silicate	8	No	No	No	12627-14-4
	Molybdenum Disulfide	28.5	No	No	No	1317-33-5
	Not Specified (Water)	63.5	No	No	No	7732-18-5
SFL-9	N-Methyl-Pyrrolidone	4.7	Yes	No	Yes	872-50-4
	Not Specified (Water)	62.8	No	No	No	7732-18-5
	Molybdenum Disulfide	32.5	No	No	No	1317-33-5

APPENDIX B
FILM THICKNESS

Many solid film lubricant applications demand small tolerances for film thickness. SFL-1, -2, -3, -5, and -6 meet MIL-L-46147B's specification for film thickness. The water-based SFL-4, -7, and -9 cannot be thinned sufficiently to meet the 0.013-mm specification. SFL-3 requires three coats to meet the 0.008-mm thickness minimum. SFL-5 was not available in bulk for testing at TFLRF. The manufacturer of this product coated the panels that were used to determine film thickness. Table B-1 presents the average film of 35 film-thickness measurements taken with the procedure described in Section 3.2.

Percent Deviation was calculated by dividing the average standard deviation, σ, by the mean film thickness, μ, for each fluid.

$$\sigma^2 = \frac{(x - \mu)^2}{N}$$

$$\text{Percent Deviation} = (\sigma / \mu) * 100\%$$

Table B-1. Film Thickness Test Summary

Product	Number of Coats	Number of Measurements	Average Thickness (mm)	Percent Deviation
SFL-1	1	35	0.00689	19
SFL-2	1	35	0.00634	19
SFL-3	3	35	0.00929	26
SFL-4	1	35	0.02591	6
SFL-5	1	10	0.00701	9
SFL-6	1	35	0.00957	18
SFL-7	1	35	0.02363	11
SFL-9	1	35	0.02282	12

APPENDIX C
ENDURANCE LIFE AND LOAD CARRYING CAPACITY TESTS

The endurance life of each product was tested under the procedure described in section 3.5. The average of the four times was reported in Table 3. The third trial of SFL-2 was omitted because the run time did not correlate with the other three times.

Table C-1. Endurance Life

Product	Time (1)	Time (2)	Time (3)	Time (4)	Average	Percent Deviation
SFL-1	12:39	11:09	12:12	12:31	12:14	5
SFL-2	5:58	7:21	Omitted	8:16	7:23	15
SFL-4	3:38	3:21	1:34	2:31	2:31	28
SFL-5	3:17	3:38	4:09	3:20	3:41	11
SFL-7	15:46	16:18	30:15	22:20	21:10	31
SFL-9	2:42	7:50	5:52	6:29	5:44	38

*Run #3 was omitted due to excessive run time.

The load carrying capacity of each product was tested under the procedure described in section 3.5. The average of the two tests was reported in Table 3.

Table C-2. Load Carrying Capacity

Product	Test 1	Test 2	Average
SFL-1	1750	2000	1875
SFL-2	1250	1000	1125
SFL-4	1000	1000	1000
SFL-5	750	1000	825
SFL-7	1470	1470	1470
SFL-9	1470	1170	1320

APPENDIX D
PRODUCTS NOT TESTED

The form letter discussed in Section 2 resulted in a large number of contacts. Information on all of the products was gathered before samples were collected. Many products were not tested because product information, such as MSDS sheets, indicated hazardous components or high VOC contents. Others were not tested because the manufacturer failed to return contacts. Graphite products were avoided because they are ineffective in inhibiting corrosion. No tungsten disulfide based samples were tested at TFLRF, because the lubricants either contained graphite or the manufacturers failed to reply to SwRI contacts. Literature indicates that these products may be more effective in both endurance life and corrosion resistance than molybdenum disulfide lubricants. Pure tungsten disulfide costs nearly four times more than pure molybdenum disulfide. Table D-1 presents the name of the manufacturer, product description and the reason the omission of the products not tested.

Table D-1. Products Not Tested

Manufacturer	Product Description	Reason for Omission
Cerac	MoS2 and WS2 aerosol	High VOC and Methylene Chloride, which is a carcinogen and on the EPA toxic chemicals list
Dicrorite Dry Lube	WS2 in lamellar form, air dry	Dicrorite failed to reply to SwRI contacts
Sentinel	MoS2 dry film lubricant	Contains 5% toluene, EPA 17 Hazardous Chemicals List #3
Micro Surface Corp.	WS2 dry film	Contains graphite, which causes corrosion
Whitford	PTFE/MoS2 products	High VOC
Kano Laboratories	MoS2 dry film lube	Contains Trichloroethylene, which is on the EPA toxic chemicals list
Zip Chem	Graphite SFLs	Contains Graphite, Corrodes
Micro Surface	MoS2/Graphite Lubricant	Contains Graphite, Corrodes and Micro Surface would not return SwRI contacts

APPENDIX E
LIQUID CARBON DIOXIDE

Carbon dioxide exists as a liquid at high pressure, 5 to 1000 atm, and a wide temperature range, from –57°C to 30°C (see Figure E-1). Supercritical liquid exists at higher pressure and temperature, but state boundries are not clearly defined in this region above the critical state. If liquid CO_2 could be used to dissolve the product's binder, this fluid could be used as both a solvent and spraying medium. That is, the pressurized liquid lubricant could be released through a spraying nozzle. The CO_2 would quickly vaporize, and the bonded lubricant would be cured in seconds. This process holds substantial advantages over the current SFL application process. First, a CO_2 based lubricant would emit no VOCs. Second, the time needed to lubricate a part would decrease dramatically. The part could be lubricated and used just seconds later, rather than 24 hours. Also, more-portable, all-in-one spraying containers could be developed for field use.

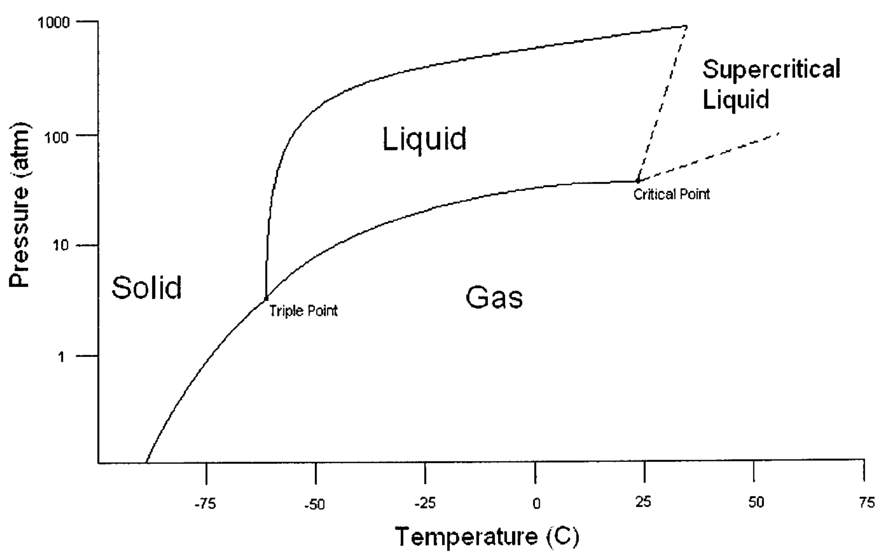

Figure E-1: Carbon Dioxide Pressure vs. Temperature Diagram